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METHOD OF TREATING FIBERS FOR PAPER-MAKING AND OTHER USES

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In preparing vegetable fibers of a woody character, such as spruce, hemlock, flax straw, and bagasse, for paper-making, it is common to treat the woody material with certain chemicals to remove the ligneous matter enveloping the fibers in a natural state. This chemical treatment is sometimes technically referred to as "cooking" the fibers.

When the fibers are overcooked, they are converted into oxy-cellulose unsuitable for paper-making, whereas, when properly cooked, they constitute a true cellulose highly valuable for paper-making and other purposes. In all known processes for cooking fibers some of the fibers are overcooked and reduced to oxy-cellulose, while others, properly cooked, remain as true cellulose. The two (oxy-cellulose and true cellulose) are frequently so intermingled that they cannot be properly separated in making paper. This results in a deterioration of the whole to the extent of the amount of the undesirable oxy-cellulose present.

One of the objects of the present invention is to overcome the objections due to oxy-cellulose, whether alone or mixed with true cellulose. A further object is to improve the character of the cellulose fibers themselves.

Many woody fibers, after being cooked to remove the ligneous matter in which they are enveloped, are, in the practical paper-making art, submitted to a bleaching action, chlorine gas being an agent frequently employed for this purpose. But, in practical operations, it has been found that this bleaching operation by the use of chlorine gas produces two highly undesirable results, that is to say, it causes a weakening of the fibers and also a shrinkage of the fibers sometimes amounting to as much as 10%.

A further object, therefore, of the present invention is to provide a method whereby woody fibers may be bleached by the use of chlorine gas or its equivalent without weakening the fibers, and whereby the shrinkage, above indicated, may be largely decreased.

With these and other objects in view, cooked fibers (oxy-cellulose, or true cellulose with more or less oxy-cellulose mixed therewith) are placed in a digester in the presence of a catalytic agent, such as nickel and zinc sulphates dissolved in water. The fibers are treated in the digester until they are thoroughly impregnated with the catalytic solution. Said impregnation may be secured either at atmospheric pressure or above atmospheric pressure. If the impregnation is to occur above atmospheric pressure, the digester is closed and the pressure therein raised to, say 100 pounds, and maintained until the fibers are thoroughly impregnated with the catalytic solution. If the pressure within the digester is raised by the application of heat thereto, steam will be formed in the digester, and after the fibers are thoroughly impregnated with the catalytic solution the pressure and steam in the digester is released, as by a suitable blow-out valve, or otherwise. After the fibers are impregnated with the catalytic solution care is taken to have sufficient water in the digester to cover the fibers. If necessary sufficient water is added for this purpose, the added water being preferably preheated, so that the contents of the digester after water is added will have a temperature of approximately 225° F. At this point a small amount each of kieselguhr, calcium carbonate and magnesium carbonate is then added as carrying agents for the nickel and zinc salts, and hydrogen gas is introduced into the digester near the bottom thereof, and permitted to rise freely through the mass, the supply of gas being continued until foaming ceases. When this point is reached the fibers are separated from the solution and thoroughly washed, and are then ready for use in making paper. The introduction of the hydrogen gas into the digester may be effected either at atmospheric pressure or with the pressure in the digester raised a few pounds (say from 5 to 10 pounds) above atmospheric pressure. In some instances it will be found that the more beneficial results are secured when the hydrogen gas in introduced under such pressure. In either case, care should be taken to recover the hydrogen gas that escapes above the surface of the fibers in the digester and store the same for reuse.

When fibers are treated as thus far de-

scribed, it will be found that they are of quality superior to that of the same fibers without such treatment, and it will be found that even when approximately pure oxy-cellulose fibers (that is fibers so far overcooked that the entire mass is free or nearly free of true cellulose) have been so treated they can be used for making paper of approximately the same quality as that obtained from the treated true cellulose fibers, and of a quality superior to that obtained through a mixture of true cellulose and oxy-cellulose fibers that have not been so treated.

Fibers treated according to the foregoing description will not be bleached. When it is desired to bleach the fibers, a small quantity of sodium perborate is added to the catalytic solution, the fibers placed therein and chlorine gas or its equivalent is then introduced into the catalytic solution with the fibers therein before the introduction of the hydrogen gas, and after a short interval of time, the hydrogen gas is then introduced. It will be found in this case that the fibers not only have their quality as paper-making fibers very decidedly increased, but that they will be bleached to a clear white.

While the exact method of procedure and quantities of material used may vary within limits, the following has been found to give excellent results:

For each 100 pounds of pulp (whether a mixed mass of oxy-cellulose and true cellulose or the oxy-cellulose alone) to be treated, 8 ounces of nickel sulphate and 1 ounce of zinc sulphate are dissolved in 500 pounds of water in an ordinary digester, and the pulp is placed therein. If the impregnation of the fibers with the catalytic solution is to be secured under pressure the digester is closed and the pressure raised in the digester (as by the application of heat) until such pressure reaches approximately 100 pounds, and the digester is allowed to stand for approximately 15 minutes. The pressure is then released from the digester, as by means of a suitable blow-off valve. Sufficient hot water is then introduced into the digester so that the solution therein will completely cover the pulp and be at a temperature of approximately 225° F. There is then introduced into the solution 2 ounces kieselguhr (infusorial earth), 4 ounces magnesium carbonate, and 2 ounces calcium carbonate. The kieselguhr, magnesium carbonate and calcium carbonate may be introduced into the solution without increasing the pressure in the digester above atmospheric pressure. Assuming that it is not intended to bleach the fibers, hydrogen gas is then introduced at or near the bottom of the digester through an inserted tube, or otherwise, and this introduction of hydrogen continued until foaming ceases, the pressure in the digester being maintained during the introduction of the hydrogen either at atmospheric pressure or a few pounds (say from 5 to 10 pounds) above atmospheric pressure. The hydrogen which escapes from the top of the mass being treated is collected and carried off by any suitable means to a suitable storage reservoir. After foaming ceases from the introduction of the hydrogen, the solution is drawn out of the digester and the pulp washed, after which it is ready for use as in the ordinary practice of paper-making.

If it is desired to bleach the fibers being treated, there is introduced into the catalytic solution from 6 to 8 ounces of sodium perborate for each 500 pounds of water used in the solution and the fibers placed therein. Before the hydrogen gas is introduced, chlorine gas or its equivalent is introduced into the digester near the bottom thereof through a suitable tube or otherwise, and the introduction thereof continued for a few moments (say 3 to 5 minutes), the length of time depending somewhat on the mass of the fibers being treated, after which, the supply of chlorine gas is discontinued, and the hydrogen gas introduced, as described above.

It will be found that the fibers so treated are bleached to a clear white, that the mass of the fibers has not shrunken, and that the fibers themselves not only have not been weakened, but that the strength and quality as paper-making fibers have been very largely increased.

By the foregoing treatment there is produced a new product, that is, hydrogenized woody fibers of superior quality for paper-making, as well as chlorinized, hydrogenized woody fibers of superior strength and of a clear white, and at a cost less than that heretofore incident to the production of lower grade fibers.

What is claimed is:

1. The process of treating vegetable fibers which consists in subjecting the same to the action of hydrogen in the presence of a catalytic agent.

2. The process of treating vegetable fibers which consists in subjecting the same to the action of hydrogen in the presence of a catalytic agent under pressure.

3. The process of treating woody fibers which consists in subjecting the same to the action of hydrogen in the presence of a catalytic agent.

4. The process of treating woody fibers which consists in subjecting the same to the action of hydrogen in the presence of a catalytic agent under pressure.

5. The process of treating oxy-cellulose fibers which consists in subjecting the same to the action of hydrogen in the presence of a catalytic agent.

6. The process of treating oxy-cellulose mixed with cellulose fibers which consists in subjecting the same to the action of hydrogen in the presence of a catalytic agent.

7. The process of treating woody fibers, which consists in impregnating the fibers with a catalytic solution, and then introducing hydrogen gas into the mass while in said solution.

8. The process of treating woody fibers which consists in dissolving a catalytic agent in water, immersing the fibers therein under pressure, then releasing the pressure and introducing hydrogen gas into the mass.

9. The process of treating woody fibers which consists in dissolving a catalytic agent in water, immersing the fibers therein, whereby the fibers are impregnated with the solution, and then introducing hydrogen gas into the mass.

10. The process of treating woody fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers therein under pressure, whereby the fibers are impregnated with the solution, then releasing the pressure, and introducing hydrogen gas into the mass.

11. The process of treating woody fibers which consists in dissolving nickel and zinc salts in water, immersing the fibers in the solution under pressure, whereby the fibers are impregnated with the solution, then releasing the pressure and introducing hydrogen gas into the mass.

12. The process of treating oxy-cellulose fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers in the solution under pressure, maintaining the pressure until the fibers are impregnated with the solution, then releasing the pressure and introducing hydrogen gas into the solution while the fibers are still immersed therein.

13. The process of treating woody fibers which consists in dissolving nickel and zinc sulphates in water, in the proportions of 8 ounces of nickel sulphate and 1 ounce of zinc sulphate for each 500 pounds of water, immersing the fibers in the solution in a closed digester, applying heat thereto until the pressure in the digester reaches 100 pounds, then cutting off the heat and allowing the mass to stand under pressure until the fibers are thoroughly impregnated with the solution, then releasing the pressure and introducing hydrogen gas into the solution while the fibers are still immersed therein.

14. The process of treating woody fibers which consists in dissolving a catalytic agent in water, immersing the fibers therein, whereby the fibers are impregnated with the solution, and then introducing hydrogen gas into the mass under pressure.

15. The process of treating woody fibers, which consists in dissolving a catalytic agent in water, introducing a carrier for the catalytic agent, and then subjecting the mass to the action of hydrogen gas.

16. The process of treating woody fibers which consists in dissolving a catalytic agent in water, immersing the fibers therein under pressure, then releasing the pressure and introducing a carrier for the catalytic agent, and then introducing hydrogen gas into the mass.

17. The process of treating oxy-cellulose fibers which consists in impregnating the fibers with a nickel and zinc sulphate solution, introducing a carrier for the nickel and zinc sulphates, and then subjecting the impregnated fibers to the action of hydrogen gas.

18. The process of treating oxy-cellulose fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers therein under pressure, whereby the fibers are impregnated with the solution, then releasing the pressure, and introducing a carrier for the nickel and zinc sulphates, and then introducing hydrogen gas into the mass.

19. The process of treating oxy-cellulose fibers which consists in impregnating the fibers with a nickel and zinc sulphate solution, introducing kieselguhr, magnesium carbonate and calcium carbonate into the solution and then subjecting the fibers to the action of hydrogen while immersed in said solution.

20. The process of treating oxy-cellulose fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers therein under pressure, whereby the fibers are impregnated with the solution, then releasing the pressure and introducing kieselguhr, magnesium carbonate and calcium carbonate, and then introducing hydrogen gas into the mass.

21. The process of treating oxy-cellulose mixed with cellulose fibers, which consists in impregnating the same with a catalytic solution, and then subjecting the mixture to the action of hydrogen while immersed in said solution.

22. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving a catalytic agent in water, immersing the fibers in the solution in a closed digester, then applying heat until the pressure in the digester reaches approximately 100 pounds, then shutting off the heat for approximately 15 minutes, then releasing the steam and pressure from the digester, adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution, and then introducing hydrogen gas into the mass until foaming ceases.

23. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving nickel and zinc sulphates in water, impregnating the fibers with the solution, and then subjecting the fibers to the

action of hydrogen while immersed in said solution.

24. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers in the solution in a closed digester, then applying heat until the pressure in the digester reaches approximately 100 pounds, then shutting off the heat for approximately 15 minutes, then releasing the steam from the digester, adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution, and then introducing hydrogen gas into the mass until foaming ceases.

25. The process of treating woody fibers which consists in dissolving a catalytic agent in water, immersing the fibers in the solution in a closed digester, then applying heat to raise the pressure in the digester, then shutting off the heat for approximately 15 minutes, then releasing the steam and pressure from the digester, then introducing a carrier for the catalytic agent, adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution, and then introducing hydrogen gas into the mass until foaming ceases.

26. The process of treating oxy-cellulose fibers which consists in impregnating the fibers with a catalytic solution, adding a carrier for the catalytic agent in said solution, then bringing the temperature of the solution to approximately 225° F., and then subjecting the fibers to the action of hydrogen while the same are immersed in said solution.

27. The process of treating oxy-cellulose fibers which consists in dissolving a catalytic agent in water, immersing the fibers in the solution in a closed digester, then applying heat to raise the pressure in the digester, then shutting off the heat for approximately 15 minutes, then releasing the steam from the digester, then introducing kieselguhr, magnesium carbonate and calcium carbonate into the solution, then adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution, and then introducing hydrogen gas into the mass until foaming ceases.

28. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers in the solution in a closed digester, then applying heat to raise the pressure in the digester, then shutting off the heat for approximately 15 minutes, then releasing the steam and pressure from the digester, then introducing a carrier for the nickel and zinc sulphates, adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution, and then introducing hydrogen gas into the mass until foaming ceases.

29. The process of treating woody fibers which consists in dissolving nickel and zinc sulphates in water, immersing the fibers in the solution in a closed digester, then applying heat until the pressure in the digester reaches approximately 100 pounds, then shutting off the heat for approximately 15 minutes, then releasing the steam and pressure from the digester, then introducing kieselguhr, magnesium carbonate and calcium carbonate into the solution, then adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution, and then introducing hydrogen gas into the mass until foaming ceases.

30. The process of treating vegetable fibers which consists in successively subjecting the same to the action of chlorine gas and hydrogen in the presence of a catalytic agent.

31. The process of treating vegetable fibers which consists in successively subjecting the same to the action of chlorine gas and hydrogen in the presence of a catalytic agent under pressure.

32. The process of treating woody fibers which consists in successively subjecting the same to the action of chlorine gas and hydrogen in the presence of a catalytic agent.

33. The process of treating woody fibers which consists in successively subjecting the same to the action of chlorine gas and hydrogen in the presence of a catalytic agent under pressure.

34. The process of treating oxy-cellulose fibers which consists in successively subjecting the same to the action of chlorine gas and hydrogen in the presence of a catalytic agent.

35. The process of treating oxy-cellulose mixed with cellulose fibers which consists in successively subjecting the same to the action of chlorine gas and hydrogen in the presence of a catalytic agent.

36. The process of treating woody fibers which consists in impregnating the fibers with a catalytic solution, and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution.

37. The process of treating woody fibers which consists in dissolving a catalytic agent in water, immersing the fibers therein under pressure, then releasing the pressure, and successively introducing chlorine gas and hydrogen gas into the mass.

38. The process of treating woody fibers

which consists in dissolving nickel and zinc sulphates in water, immersing the fibers therein under pressure, whereby the fibers are impregnated with the solution, then releasing the pressure and successively introducing chlorine gas and hydrogen gas into the mass.

39. The process of treating woody fibers which consists in dissolving nickel and zinc salts in water, immersing the fibers in the solution under pressure, whereby the fibers are impregnated with the solution, then releasing the pressure and successively introducing chlorine gas and hydrogen gas into the mass.

40. The process of treating woody fibers which consists in impregnating the fibers with a catalytic solution containing a carrier for the catalytic agent, and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution.

41. The process of treating woody fibers which consists in impregnating the fibers with a catalytic solution containing sodium perborate and a carrier for the catalytic agent, and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution.

42. The process of treating woody fibers which consists in dissolving a catalytic agent in water, adding sodium perborate to the solution, then immersing the fibers under pressure in said solution, then releasing the pressure and successively introducing chlorine gas and hydrogen gas into the mass.

43. The process of treating woody fibers which consists in dissolving a catalytic agent in water, adding sodium perborate to the solution, then immersing the fibers in said solution under pressure of approximately 100 pounds, then reducing the pressure to from 5 to 10 pounds and successively introducing chlorine gas and hydrogen into the mass.

44. The process of treating woody fibers which consists in dissolving a catalytic agent in water, introducing sodium perborate therein, then subjecting the fibers in the solution to pressure of approximately 100 pounds, then reducing the pressure to from 5 to 10 pounds, then successively introducing chlorine gas and hydrogen gas into the mass, then removing the fibers from the solution and washing the fibers.

45. The process of treating woody fibers which consists in impregnating the fibers with a solution of nickel and zinc sulphates and sodium perborate and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution.

46. The process of treating oxy-cellulose fibers which consists in dissolving nickel and zinc sulphates in water, adding to the solution approximately the same quantity of sodium perborate as of the nickel and zinc sulphates combined, then immersing the fibers in the solution under pressure, maintaining the pressure until the fibers are impregnated with the solution, and then reducing the pressure to from 5 to 10 pounds and successively introducing chlorine gas and hydrogen gas into the solution while the fibers are still immersed therein.

47. The process of treating woody fibers which consists in dissolving nickel and zinc sulphates and sodium perborate in water in the proportions of 8 ounces of nickel sulphate, one ounce zinc sulphate and approximately 9 ounces of sodium perborate to each 500 pounds of water, immersing the fibers in the solution in a closed digester, applying heat thereto until the pressure in the digester reaches 100 pounds, then cutting off the heat and allowing the mass to stand under pressure until the fibers are thoroughly impregnated with the solution, then reducing the pressure to from 5 to 10 pounds and successively introducing chlorine and hydrogen gas into the solution while the fibers are still immersed therein.

48. The process of treating woody fibers which consists in impregnating the fibers with a solution of nickel and zinc sulphates and sodium perborate, and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution while maintaining the solution at approximately 225° F.

49. The process of treating woody fibers which consists in dissolving a catalytic agent together with a like amount of sodium perborate in water, impregnating the fibers with the solution, and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution.

50. The process of treating woody fibers which consists in dissolving a catalytic agent and a like amount of sodium perborate in water, adding a carrier for the catalytic agent, impregnating the fibers with the solution, and then successively subjecting the fibers to the action of chlorine gas and hydrogen while immersed in said solution.

51. The process of treating woody fibers which consists in dissolving a catalytic agent together with a like amount of sodium perborate in water, immersing the fibers therein under pressure, then releasing the pressure to near atmospheric pressure and introducing a carrier for the catalytic agent and then successively introducing chlorine and hydrogen gas into the mass.

52. The process of treating oxy-cellulose fibers which consists in dissolving nickel and zinc sulphates and a like amount of sodium perborate in water, immersing the fibers therein under pressure whereby the fibers are impregnated with the solution, then reducing the pressure, introducing a carrier for the

nickel and zinc sulphates and then successively introducing chlorine and hydrogen gas into the mass.

53. The process of treating oxy-cellulose fibers which consists in dissolving nickel and zinc sulphates and a like amount of sodium perborate in water, immersing the fibers under pressure whereby the fibers are impregnated with the solution, then releasing the pressure and introducing kieselguhr, magnesium carbonate and calcium carbonate into the solution and then successively introducing chlorine and hydrogen gas into the mass of fibers while in said solution.

54. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving a catalytic agent and a like amount of sodium perborate in water, immersing the fibers in the solution in a closed digester, and then applying heat until the pressure in the digester reaches approximately 100 pounds, and then shutting off the heat for approximately 15 minutes, and then releasing steam and pressure from the digester, adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to insure complete immersion of the fibers in the solution and successively introducing chlorine and hydrogen into the mass until foaming ceases.

55. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving nickel and zinc sulphates and a like amount of soduim perborate in water, immersing the fibers in the solution in a closed digested, and then applying heat until the pressure in the digester reaches approximately 100 pounds, and then shutting off heat for approximately 15 minutes, and then releasing the steam from the digester, adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to secure complete immersion of the fibers in the solution and then successively introducing chlorine and hydrogen gas into the mass until foaming ceases.

56. The process of treating woody fibers which consists in dissolving a catalytic agent and a like amount of sodium perborate in water, immersing the fibers in the solution in a closed digester, and then applying heat to raise the pressure in the digester, then shutting off the heat, then releasing the steam and pressure from the digester, then introducing a carrier for the catalytic agent, adding water at a temperature to bring the temperature of the mass in the digester to 225° F. and in quantity to insure complete immersion of the fibers in the solution, then introducing chlorine gas into the mass and thereafter introducing hydogen gas into the mass until foaming ceases.

57. The process of treating oxy-cellulose fibers which consists in dissolving a catalytic agent and approximately a like amount of sodium perborate in water, immersing the fibers in the solution in a closed digester and raising the pressure in the digester, releasing the pressure in the digester, then introducing kieselguhr, magnesium carbonate and calcium carbonate into the solution, then adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity to secure complete immersion of the fibers in the solution, then introducing chlorine gas into the mass, and then raising the pressure in the digester to approximately 4 pounds and introducing hydrogen gas into the mass until foaming ceases.

58. The process of treating oxy-cellulose mixed with cellulose fibers which consists in dissolving nickel and zinc sulphates and a like amount of sodium perborate in water, immersing the fibers in the solution in a closed digester, and applying heat to raise the pressure in the digester, then shutting off the heat for approximately 15 minutes, then releasing steam and pressure from the digester, then introducing a carrier for nickel and zinc sulphates, adding water at a temperature to bring the temperature of the mass in the digester to 225° F. and in quantity to secure complete immersion of the fibers in the solution, and then introducing chlorine gas into the mass, and then introducing hydrogen gas into the mass under a pressure of approximately 4 pounds and continuing the supply of hydrogen until the foaming ceases.

59. The process of treating woody fibers which consists in dissolving nickel and zinc sulphates and a like amount of sodium perborate in water, immersing the fibers in the solution in a closed digester, then applying heat until the pressure in the digester reaches approximately 100 pounds, then shutting off the heat for approximately 15 minutes, then releasing steam and pressure from the digester, then introducing kieselguhr, magnesium carbonate and calcium carbonate into the solution, then adding water at a temperature to bring the temperature of the mass in the digester to approximately 225° F. and in quantity sufficient to secure complete immersion of the fibers in the solution, and then introducing chlorine gas in the mass and thereafter introducing hydrogen gas into the mass under pressure and until foaming ceases.

60. A mass of chlorinized and hydrogenized woody fibers.

In testimony whereof I have signed this specification.

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